

(19) World Intellectual Property Organization  
International Bureau



(43) International Publication Date  
29 March 2001 (29.03.2001)

PCT

(10) International Publication Number  
**WO 01/21740 A1**

- (51) International Patent Classification<sup>7</sup>: **C10M 125/06**, 129/68
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- (21) International Application Number: **PCT/US00/26167**
- (81) Designated States (*national*): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.
- (22) International Filing Date:  
22 September 2000 (22.09.2000)
- (25) Filing Language: English
- (26) Publication Language: English
- (84) Designated States (*regional*): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).
- (30) Priority Data:  
60/155,345 22 September 1999 (22.09.1999) US
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**Published:**

*With international search report.*

*For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.*

**WO 01/21740 A1**

(54) Title: **NON-STAINING, ACTIVE METAL-WORKING FLUID**

(57) Abstract: Non-staining, active metal-working compositions are disclosed. The compositions contain active sulfur to provide extreme pressure properties for metal-working fluids. A metal corrosion inhibitor is disclosed that reduces the corrosivity of free sulfur on non-ferrous metallic objects.

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NON-STAINING, ACTIVE METAL-WORKING FLUID

5

BACKGROUND OF THE INVENTION

Field of the Invention

The present invention relates generally to compositions for lubricating articles in metalworking operations. More particularly the present invention relates  
10 to lubricating compositions, which are non-corrosive and non-staining to non-ferrous metals.

Brief Description Of Related Technology

15 Metalworking processes mechanically shape and work metallic articles or work pieces. Lubricating fluids are often used on the work pieces in metalworking processes to reduce friction between a tool and the work piece and to dissipate heat resulting from any remaining friction. The

reduction of friction and dissipation of heat promotes tool life, increases production and allows the attainment of high quality finished metal products.

5 Metalworking operations mechanically shape and work metallic work pieces by cutting and non-cutting operations. The cutting processes include, for instance, drilling, grinding, milling, tapping, turning and broaching. Non-cutting processes include, for example, rolling, drawing, extrusion, drawing and ironing, punching, stamping and spinning  
10 processes. These metal working processes are often characterized into three general categories (for instance, light duty, medium duty and heavy duty) according to severity of the operation. Light duty jobs may include boring and milling. Medium duty may include tapping, reaming and gear  
15 cutting. Heavy duty may include broaching and threading. Increased friction and increased heat generation generally coincide with increased severity or duty of a particular metalworking process.

20 The type of metal also often influences the duty of the metalworking operation. For example, a metalworking operation on a stainless steel is often a more severe, operation than a similar operation on a carbon steel due to the higher strength of the stainless steel.

25 Lubricating fluids for metalworking operations are also distinguished by duty corresponding to a particular metalworking operation. A light duty fluid is generally appropriate for lubricating light duty metalworking operations. A heavy duty fluid has greater lubricity characteristics than a light duty fluid and is generally  
30 appropriate for lubrication in a heavy duty operation. Increased lubricity of a heavy duty fluid is often partially achieved through the use of a more viscous oil than used for light duty fluid.

35 Additives may also be incorporated into a heavy duty metalworking fluid to increase the lubricity of the fluid at metal-to-metal contact points, such as at points where a tool contacts a work piece. Sulfur is a common additive used to increase lubricity at metal-to-metal contact points. Many

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fluids would not function as a heavy duty metalworking lubricant without the use of a sulfur additive.

A heavy duty lubricant containing sulfur, however, may not always be appropriate for the lower duty operations or heavy duty operations with particular work pieces. While a sulfur-containing heavy duty fluid can generally provide adequate lubricity for light, medium and heavy duty metalworking operations, sulfur-containing heavy duty fluids often stain or corrode non-ferrous metals. As such, there is a need for a sulfur-containing metalworking fluid suitable for heavy-duty operations, which does not stain or corrode nonferrous metals.

#### SUMMARY OF THE INVENTION

The present invention provides compositions for heavy duty metalworking fluids that do not corrode or severely stain nonferrous metals. The inventive compositions also provide greater lubricity for metalworking processes using ferrous and nonferrous metals. Improved product finishes result from the use of the inventive compositions on both ferrous and nonferrous metallic articles.

In one aspect the present invention provides a metalworking fluid composition that does not stain non-ferrous metals and is useful for heavy duty metalworking operations thereon. In one desirable feature the present inventions contains greater quantities sulfur than previously achieved in the prior art to increase lubricity and wear characteristics of a metalworking oil without staining nonferrous articles.

In another aspect of the present invention lubricating compositions are provided which include a fatty oil to inhibit staining of non-ferrous metals during metalworking processing. Useful fatty oils include monoglycerides, diglycerides, triglycerides, esters of monocarboxylic acids, esters of dicarboxylic acids and combinations thereof. The fatty oils may also be sulfurized, chlorinated or chlorosulfurized.

The invention will now be described with reference to the section entitled "Detailed Description of the Invention."

#### DETAILED DESCRIPTION OF THE INVENTION

5

The present invention relates to metalworking fluid compositions suitable for heavy duty metalworking processes without the disadvantages described above. A metalworking lubricant serves many functions, including use as a heat-  
10 transfer medium, providing protection against rust and corrosion, and serving as a carrier for debris. Among the many other advantages and uses of the inventive metalworking compositions are (1) preventing corrosion and staining of the tool and the work piece; (2) keeping a metalworking tool cool  
15 and preventing it from being heated to a temperature at which the hardness and resistance to abrasion are reduced; (3) maintaining the work piece cool, thereby preventing it from being machined in a warped shape to inaccurate final dimensions; (4) providing a good finish on the work piece; (5)  
20 aiding in satisfactory chip formation to promote the metalworking operation; (6) washing away chips, especially in deep-hole drilling, milling and grinding; (7) lubricating moving machine parts close to the cutting tool; and (8) reducing power consumption of the metalworking operation  
25 through lubrication.

In one aspect the inventive composition is an oil-based lubricant. An oil-based fluid composition is particularly useful in metalworking operations where lubrication and high grade finishing cuts are especially  
30 desirable, or where aqueous fluids adversely effect product finishes. For example, frictional heat from the metalworking operation is dissipated in an oil film resulting from application of the oil onto a work piece. The amount of heat dissipated is generally related to the film thickness, fluid  
35 velocity and fluid density. Insufficient heat dissipation can often cause high fluid temperatures that decrease the viscosity of the oil sufficiently to break down the film.

Such a film breakdown can result in metal-to-metal contact between the tool and the work piece. Metal-to-metal contact can often result in failure of the tool, the work piece, or both the tool and the work piece.

5           Thus, film thickness, fluid density, velocity and viscosity are important properties for such an oil-based lubricating composition. High severity or heavy-duty metalworking operations generally require oil-based lubricants with a high viscosity to provide adequate lubrication. Oil-  
10 based lubricants may be broadly classified into duties based on their viscosity. The viscosity ranges described herein are not intended to limit the scope of the invention, but are generally recognized in the industry and are intended to serve as examples to aid in the understanding of the invention. A  
15 light duty fluid generally has a kinematic viscosity from about 10 cSt (centistokes) at 25°C to about 30 cSt at 25°C. A medium duty fluid generally has a kinematic viscosity from about 30 cSt at 25°C to about 75 cSt at 25°C. A heavy-duty fluid generally has a kinematic viscosity greater than 75 cSt  
20 at 25°C. A heavy-duty fluid which has a maximum kinematic viscosity of 160 cSt at 25°C is particularly useful as a heavy duty metalworking fluid.

          In another aspect of the present invention, an inventive composition which serves as a heavy duty  
25 metalworking fluid with a kinematic viscosity range of about 75 cSt to about 160 cSt at 25°C is useful. Desirably, compositions of the present invention may have a kinematic viscosity range of about 75 cSt to about 90 cSt at 25°C; and more desirably may have kinematic viscosity ranges of about 20  
30 cSt to about 60 cSt at 40°C and about 4 cSt to about 8 cSt at 100°C.

          The viscosity of the lubricating oil used in the inventive compositions may be selected by choosing an appropriate base oil or by mixing various base oils.  
35 Appropriate base oils include lubricating oil fractions of naphthenic, paraffinic or naphthenic/paraffinic petroleum. These lubricating oil fractions may be unrefined, acid-refined, solvent-refined, hydrotreated or hydrocracked as

required by the particular lubricating need. Lubricating oil fractions and hydrotreated or hydrocracked oil fractions obtained from vacuum distillation of petroleum are also useful.

5           Mixing of various base oils may also be useful for obtaining a desired viscosity of the inventive composition.

          Among useful base oils are lubricating oil fractions of International Standards Organization (ISO) grade numbers 22, 32, 46, 68 and combinations thereof. The ISO grade  
10 numbers are approved for classifying industrial lubricants according to a mid-point of a viscosity range expressed in centistokes at 40°C as shown below in Table 1. Other ISO grade numbers are also useful with the practice of the present invention.

Table 1		
ISO Viscosity Grade Number	Viscosity Grade Ranges in Centistokes at 40°C	
	Minimum	Maximum
22	19.8	24.2
32	28.8	35.2
46	41.4	50.6
68	61.2	74.8

15           Useful base oils may also include oils from animals, oils from plants, synthetic oils and combinations thereof. Oils of lubricating viscosity derived from coal, shale or tar sands are also useful.

20           Useful synthetic lubricating oils include, without limitation, hydrocarbon oils and halo-substituted hydrocarbon oils such as polymerized and interpolymerized olefins; alkylbenzenes; polyphenyls; alkylated diphenyl ethers and alkylated diphenyl sulfides. Another useful class of useful  
25 synthetic lubricating oils includes the esters of dicarboxylic acids of relatively low acid number, for instance dibutyl adipate, di(2-ethylhexyl) sebacate, di-n-hexyl fumarate, dioctyl sebacate, diisooctyl azelate, diisodecyl azelate, dioctyl phthalate, didecyl phthalate, and dieicosyl sebacate.  
30 Esters useful as synthetic oils also include those made from C<sub>5</sub>

to C<sub>12</sub> monocarboxylic acids, polyols and polyol ethers such as neopentyl glycol, trimethylolpropane, pentaerythritol, dipentaerythritol and tripentaerythritol.

5 Metalworking lubricants, especially heavy duty lubricants, are also often distinguished as being suitable or unsuitable for extreme pressure applications. An extreme pressure lubricant is a lubricant that prevents sliding metal surfaces from seizing under extreme pressure conditions. The  
10 seizing of metal surfaces result from friction between opposing asperities. Asperities are microscopic projections on metal surfaces resulting from metalworking operations. Interference between opposing asperities in sliding or rolling applications is a source of friction and can lead to metal welding and scoring.

15 One technique for measuring extreme pressure properties of a lubricant is to measure a load force between sliding surfaces which can be sustained by lubricant without seizing of the sliding surfaces. Such a technique is described as a Falex load test, which is an ASTM standard test  
20 for fluid lubricants (ASTM D 3233). As used herein the phrase "extreme-pressure composition" and its variants refer to a composition that has a Falex reference load of 1,000 pounds force or greater. The Falex load test is further described herein in conjunction with Example 2.

25 Typically a lubricant additive is incorporated into an appropriate base oil to obtain a lubricant that prevents sliding metal surfaces from seizing under conditions of extreme pressure (EP). At the local high temperatures associated with metal-to-metal contact, an EP additive is  
30 believed to interact with the metal to form a surface film that prevents the welding of opposing asperities, and the consequent scoring or seizing that is destructive to sliding surfaces under high loads. Compounds of sulfur, chlorine, phosphorus and combinations thereof are useful as EP additives  
35 with the present invention. In one aspect of the present invention, the lubricant contains from 0 to about 3 weight percent chlorine, from 0 to about 2 weight percent phosphorus



and from 0 to about 4 weight percent chemical bound sulfur, such as the sulfur contained in t-dodecyl polysulfide.

Metalworking lubricants that contain EP additives are typically classified as "active" as compared to lubricants without EP additives, which are typically classified as "inactive". As used herein the term "active" and its variants refer to an additive or a fluid that is stable at room temperatures but provides necessary protection against metal seizing, galling or scoring in the high-friction, high-temperature metal-to-metal contact areas.

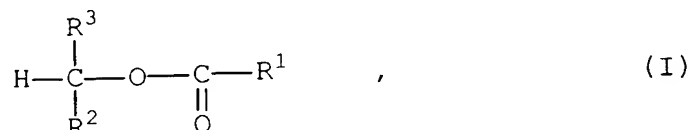
In a further aspect of the present invention, active sulfur is included to provide adequate lubrication at extreme pressure conditions. The sulfur is combined with the lubricating oil by sulfurizing techniques which include contacting an oil and a sulfur compound at high temperatures under an inert atmosphere. Sulfur, which when contacted with the oil, reacts with the oil and becomes chemically bound by the oil molecules is referred to as inactive or reacted sulfur. Unreacted sulfur is sulfur which for instance when heated with stabilizing oil is held in solution and is not chemically bound by the oil molecules. The unreacted or free sulfur is the component which provides the extreme pressure and metal cutting lubricant properties. The unreacted sulfur may include those sulfur atoms bound by sulfur-to-sulfur bonds. As used herein the phrase "free sulfur" and its variants refer to sulfur in a lubricating fluid composition which reacts with metallic copper at a temperature of 149°C (300°F) as prescribed in ASTM D 1662 test method. A desirable free sulfur is Elco 240, available from the Elco Corporation of Cleveland, Ohio.

Although free sulfur is active for extreme pressure lubricants in metalworking processes, free sulfur often corrodes nonferrous articles. Thus, in another aspect of the present invention, the lubricating composition includes one or more materials that inhibit the corrosive effects of free sulfur, without inhibiting or otherwise detracting from the extreme pressure lubricating effects of free sulfur. Fatty oils having monoglycerides, diglycerides, triglycerides,

esters of monocarboxylic acids and esters of dicarboxylic acids are useful as such materials and serve to inhibit the corrosive effects of free sulfur, while maintaining the sulfur active for extreme pressure lubrication. The fatty oils present from 5 to 30 volume percent of the lubricating composition are useful with the present invention. While not intended to be bound by a particular theory, one possible explanation for these beneficial effects of incorporating fatty oils may be due to their polar nature which results in an attraction of the fatty oil to the metallic surface thereby providing a barrier against corrosive metal-to-sulfur bonding.

The availability of free sulfur, proximal to the metallic surface is believed to provide extreme pressure lubricating properties to the composition, without the deleterious corrosion effects frequently caused by sulfur on metallic surfaces.

Fatty oils useful with the present invention include glycerides having the following formula:

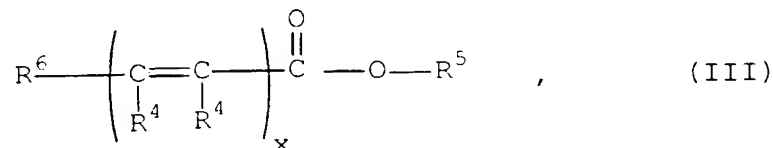


where  $\text{R}^1$  is a saturated or unsaturated  $\text{C}_3$  to  $\text{C}_{24}$  aliphatic hydrocarbon, and  $\text{R}^2$  and  $\text{R}^3$  are the same or different and can be hydrogen or



where  $\text{R}^1$  is as defined above.

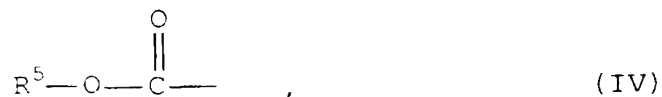
Fatty oils useful with the present invention also include esters of carboxylic acids having the following formula:



where  $\text{R}^4$  is hydrogen or a saturated or unsaturated  $\text{C}_3$  to  $\text{C}_{12}$

aliphatic hydrocarbon,  $\text{X}$  is 1, 2 or 3,  $\text{R}^5$  is a saturated or

unsaturated C<sub>3</sub> to C<sub>24</sub> aliphatic hydrocarbon, and R<sup>6</sup> is defined by the following formula:



where R<sup>5</sup> is as defined above. Desirably the base oil is a  
5 hydrotreated naphthenic oil, such as Chevron Metalworking Fluid Grade 45A, available from Chevron Products Company, San Francisco, California, which includes about 18 volume percent fatty oil as described above.

10 The present invention has improved capability for lubricating surfaces under extreme pressure conditions. The capability to lubricate under extreme pressures is evaluated by a standard test as described in Example 2A. Lubricating capabilities that result in greater than 4,500 pounds-force (lbf.) as measured by ASTM D 3233A are useful with the present  
15 invention.

Furthermore, the present invention has improved lubricating capability for reducing wear between contacting surfaces. The lubricating capability to reduce wear is evaluated by a standard test as described in Example 2B.  
20 Lubricating capabilities that result in reduce wear of less than 10 teeth as measured by ASTM D 2670 are useful with the present invention.

Moreover, the present invention also has improved anti-wear property for surfaces in sliding contact with one  
25 and the other. The anti-wear property is evaluated by a standard test as described in Example 4. Anti-wear properties that result in an average wear scar diameter of less than 0.07 mm as measured by ASTM D 4172 are useful with the present invention.

30 The present invention is further described below in the following examples, which are intended to further elucidate the invention, and are not to be construed, in any way as limiting.

EXAMPLESExample 1: Corrosivity Tests

Active metalworking lubricating fluid compositions were prepared by combining active sulfur in the form of alkylpolysulfide into two base oil compositions. Both base oil compositions were hydrotreated lubricating oil fractions of petroleum.

The first composition represented a control lubricating composition. The base oil for this composition is a hydrotreated paraffin oil and is available commercially as Chevron Neutral Oil 100R from Chevron Products Company.

The second composition was representative of an inventive composition (inventive composition one), and contained a fatty oil characterized as a  $C_8$  to  $C_{20}$  triglyceride having a fatty oil species of  $C_{14}$  and  $C_{18}$  hydrocarbon numbers to inhibit the corrosive effects of free sulfur. The base oil of this composition was Chevron Metalworking Fluid Grade 45A. Properties of these compositions are shown below in Table 2.

Active sulfur, such as Elco 240, was added to both of these compositions. Up to about 1.4 weight percent active sulfur was added to the control composition and up to about 14.4 weight percent active sulfur was added to the inventive composition. Corrosivity of the compositions were then determined by standard test methods as described herein.

The Copper Strip Corrosion Test (ASTM D 130) determines corrosivity of lubricating oils towards nonferrous metals. In this test a copper strip is immersed into a lubricating oil composition containing the test additive. The lubricating oil composition is maintained at  $100^{\circ}\text{C}$  ( $212^{\circ}\text{F}$ ) for 2 hours. The degree of discoloration of the copper strip is obtained from an ASTM standard comparison chart. A rating of 1a indicates a very low degree of discoloration and consequently very slight corrosivity of the lubricating oil composition towards copper.

Ratings of higher numbers, such as 4a or 4b, show higher reactivity, which indicate severe corrosion of the copper strip. The results of testing the inventive composition

and the control composition in the Copper Strip Corrosion Test are also set forth in Table 2.

Table 2: Copper Strip Corrosivity Tests, ASTM D 130		
Description	Control Lubricating Composition	Inventive Lubricating Composition One
Specific Gravity	0.86 at 15.6°C	0.91 at 15°C
Kinematic Viscosity		
cSt at 25°C	34.0	86.5
cSt at 40°C	19.7	38.8
cSt at 100°C	4.0	6.2
Sulfur, Total	10 ppm	2.2 Wt.%
Sulfur, Active, ASTM D 1662	--	--
Fatty Oil, Vol.%	--	18.2
Active Sulfur, Wt% of Total Composition	Classification	Classification
0.0	1b	1b
0.4	4a	1b
0.7	4b	1b
1.1	4b	1b
1.4	4b	1b
3.6	--	2b
7.2	--	3a
10.8	--	3b
14.4	--	4b

5           The fatty oil present in the inventive composition proved effective in inhibiting corrosivity effects of free sulfur. The inventive composition with the fatty oil did not corrode the copper strip until the active sulfur was increased to about 14.4 weight percent. The control sample without the  
10 fatty oil corroded the copper strip at about 0.4 weight percent active sulfur. For reference, the classifications of

the Corrosivity Tests, Copper Strip ASTM D 130 are shown below in Table 3.

Table 3: Corrosivity Tests, Copper Strip ASTM D 130		
Classification	Designation	Description
1a	Slight Tarnish	Light Orange, almost the same as freshly polished strip
1b	Slight Tarnish	Dark Orange
2a	Moderate Tarnish	Claret red
2b	Moderate Tarnish	Lavender
2c	Moderate Tarnish	Multicolored with lavender blue or silver, or both, overlaid on claret red
2d	Moderate Tarnish	silvery
2e	Moderate Tarnish	Brassy or gold
3a	Dark Tarnish	Magenta overcast on brassy strip
3b	Dark Tarnish	Multicolored with red and green showing (peacock), but no gray
4a	Corrosion	Transparent black, dark gray or brown with peacock green barely showing
4b	Corrosion	Graphite or lusterless black
4c	Corrosion	Glossy or jet black

#### 5 Example 2: Extreme Pressure and Wear Tests for Lubricants

The inventive composition of Example 1 was tested for extreme pressure and wear properties with added active sulfur and without added active sulfur. A summary of the tests is provided below.

10

#### A. Falex Extreme Pressure Test, ASTM D 3233 (Test Method A)

The capability of lubricating oil compositions to lubricate under extreme pressures can be measured by this

test. The Falex machine is manufactured by the Falex Corporation of Aurora, IL. In this test two opposing stationary V-blocks are pressed by a nutcracker arrangement of lever arms towards each other against an interposing rotating steel pin test specimen. The rotating test specimen is driven by a chuck through a brass shear pin. The V-block and pin test specimens are immersed in a vessel of the test lubricant at a preselected temperature. The machine is operated 290 rpm and the specimens are broken in at 300 pounds-force (lbf) or 1334 Newtons (N) loading. During the test, loading between the V-blocks and the rotating pin is increased until seizure occurs or until a maximum load of 4,500 lbf (20,000 N) is applied. The failure point, if any, is indicated by shearing of the brass pin holding the rotating shaft. The load at failure in pounds is taken as a quantitative measure of the extreme-pressure properties of the oil compositions.

Mineral oils may fail at 600 to 900 lbf. Oils with moderately effective extreme-pressure additives may fail at 1,000 to 2,000 lbf and very effective extreme-pressure additives will permit loadings in excess of 4,500 lbf or no failure. The limit of the test machine is 4,500 lbf.

The results of testing oil compositions of this invention in this test are set forth in Table 4.

Table 4: Inventive Composition One, Lubricating Tests	
Active Sulfur, weight Percent of Total Composition	Falex Extreme Pressure Test (ASTM D 3233A), lbf.
0.0	2370
1.4	4500+

25

Combining active sulfur into the inventive composition increased the extreme-pressure load results from a Falex reference load of 2,370 lbf without active sulfur to a Falex reference load of 4,500+lbf with active sulfur. As used herein the phrase "Falex reference load" and its variants refer to the test results from the Falex Extreme Pressure Test

30

(ASTM D 3233A) as described herein. These results may be compared to test results on other commercially available products as set forth in Table 5a. Other commercially available products ranged from a Falex reference load of 1,270 to 4,500 +lbf when under similar conditions.

#### **B. Falex Wear Test, ASTM D 2670**

The equipment and test specimens as detailed above for ASTM D 3233 were used for Falex wear tests. After the test specimens are immersed in the lubricant, the test specimen is rotated at 290 rpm under a load of 350 lbf for a 5 minute break-in period. The test is then run for 15 minutes under constant load of 900 lbf. As wear, if any, occurs on the test specimen, the load would decrease. The load, however, is maintained constant by advancing a ratchet wheel.

The test results report the number of teeth advanced on this ratchet wheel as required to maintain the constant load during the test period. Higher reported numbers correlate to higher degrees of wear.

The results of testing oil compositions of this invention in this test are set forth in Table 5. The inventive composition without active sulfur had a Falex reference wear of six. The Falex reference wear improved to zero when tested on an inventive composition with active sulfur. As used herein the phrase "Falex reference wear" and its variants refer the test results from the Falex Wear Test (ASTM D 2670) as described herein. These results may be compared to test results on other commercially available products as set forth in Table 5a. When tested under similar conditions, these commercially available products had Falex reference wear results from 5 to 27 with one commercial product failing the test.

Table 5: Inventive Composition One, Lubricating Tests	
Active Sulfur, weight Percent of Total Composition	Falex Wear Test (ASTM D 2670), No. of Teeth.
0.0	6
1.4	0



As evidenced from Table 5, the inventive composition with active sulfur proved to an effective extreme pressure, heavy-duty metalworking fluid.

As evidenced below from Table 5a, none of the commercially available active, heavy duty metal working fluids provided exceptional wear and extreme pressure properties at the corrosivity rates of the compositions of the present invention. All of these commercially available products tested to be corrosive on copper strips as evidenced by ASTM D130 classifications of 4a to 4c.

Table 5a: Lubricating and Corrosivity Results for Commercially Available Heavy Duty, Active Metalworking Fluids				
Commercial Fluid / (Source)	Kinematic Viscosity, cSt at 25°C	Falex Wear Test (ASTM D 2670), Teeth	Falex Extreme Pressure Test (ASTM D 3233A), Lbs-f	Corrosivity, Cu Strip ASTM D 130
Tri-Cut (LPS)	133.3	27	4500+	4a
Omega (Mobil)	87.1	Fail	1270	4c
31C (Chevron)	77.8	5	1770	4b
Rapid Tap (Relton)	54.1	8	4500+	4a

### Example 3: Surface Finish

The test pins from the Falex Wear Test of Example 2 on the inventive compositions were measured for surface roughness. These test pins were standard ASTM D 2670 test pins of AISI 3135 steel with a surface finish of 5 to 10 micro inches prior to the Falex Wear Test. The surface roughness was measured by using a Surftest 211 Surface Roughness Tester, which is available from Mytutoyo Corp., located in Tokyo, Japan. The Surftest 211 measures and reports the arithmetical mean deviation of the roughness profile of a machine's surface.

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The inventive composition of example 2 which did not have active sulfur had a surface roughness of about 48 micro inches. The inventive composition of example 2 with active sulfur had a surface roughness of about 23 micro inches.

5 Thus, the inventive composition with active sulfur proved more effective in providing an improved finish on a ferrous metal work piece.

**Example 4: Four-Ball Wear Test, ASTM D 4172**

10 The capability of lubricating compositions to lubricate surfaces in sliding contact can be measured by this test. A Four-Ball Wear Test machine is utilized in this test and is manufactured by Falex Corporation of Aurora, IL. In  
15 this test, three steel balls are clamped together and covered with a lubricating composition. A fourth ball is pressed with a specified force into a cavity formed by the three clamped balls. The temperature of the lubricating composition is maintained at about 75°C while the fourth ball is rotated at  
20 1200 rpm for 60 minutes. A microscope is then used to measure the diameter of scars on the three balls. The average wear scar diameter in millimeters is reported. Lower wear scar  
25 diameters indicate better anti-wear properties of a lubricating composition in sliding contact as compared to a lubricating composition resulting in higher wear scar diameters.

The inventive composition at zero and 1.4 percent active sulfur and a commercially available heavy duty, active metalworking fluid were tested under this test condition. As evidenced from Table 6, the inventive composition had lower  
30 Four-Ball wear scar diameters as compared to the other commercially available product. As used herein the phrase "Four-Ball wear scar" and its variants refer to the test results from the Four-Ball Wear Test (ASTM D 4172) as described herein. Furthermore, active sulfur also resulted in  
35 an improvement in wear scar diameter for the inventive composition.

**Table 6:** Inventive Composition One, Four-Ball Wear Scar Tests

Lubricating Composition	Avg. Wear Scar Diameter, mm (ASTM D 4172)
Inventive Composition One at 0.0 wt.% Active Sulfur	0.067
Inventive Composition One at 1.4 wt.% Active Sulfur	0.047
Tri-Cut (LPS)	1.35

Moreover, the wear scar diameters for the inventive composition can be further improved by incorporating other additives, such as inactive sulfur, into the lubricating composition. A Four-Ball wear scar of about 0.32mm was achieved with the above inventive composition at 1.4 weight percent active sulfur with about 3 weight percent inactive sulfur (t-dodecyl-polysulfide).

While there have been described various aspects of the present invention, those skilled in the art will realize that various aspects and embodiments can be made without departing from the spirit of the present invention, and it is intended all such further modifications and changes be included within the scope of the claims.

What Is Claimed Is:

1. A composition for lubricating metallic work pieces comprising:

(a) an oil having a viscosity of about 75 cSt to about 160 cSt at 25°C;

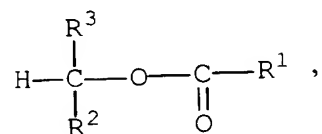
(b) free sulfur in an amount sufficient to provide lubrication; and

(c) a metal corrosion inhibitor to prevent corrosion of said work pieces,

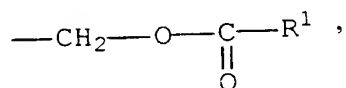
wherein lubrication is demonstrated by measurements selected from the group consisting of Falex reference wear, Falex reference load, Four-Ball wear scar diameter, and combinations thereof.

2. The composition of Claim 1, wherein said composition is a metalworking composition.

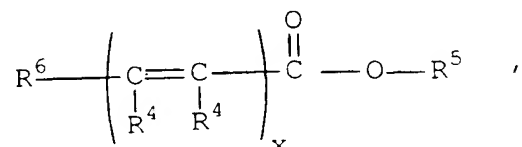
3. The composition of Claim 1, wherein said metal corrosion inhibitor is a fatty oil selected from the group consisting of a glyceride, an ester of a carboxylic acid, and combinations thereof, wherein said glyceride is represented by the formula of



wherein R<sup>1</sup> is a saturated or unsaturated C<sub>3</sub> to C<sub>24</sub> aliphatic hydrocarbon, and R<sup>2</sup> or R<sup>3</sup> is hydrogen or

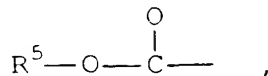


wherein R<sup>1</sup> is as defined above, and said ester is represented by the formula of



wherein R<sup>4</sup> is hydrogen or a saturated or unsaturated C<sub>3</sub> to C<sub>12</sub> aliphatic hydrocarbon, X is 1, 2 or 3, R<sup>5</sup> is a saturated or

unsaturated C<sub>3</sub> to C<sub>24</sub> aliphatic hydrocarbon, and R<sup>6</sup> is represented by the formula of



wherein R<sup>5</sup> is as defined above.

4. The composition of Claim 1, wherein said fatty oil is about 5 to about 30 volume percent based on said composition.

5. The composition of Claim 1, wherein said sulfur is present in amounts of from about 0.4 to about 12 percent by weight of said composition.

6. The composition of Claim 1, wherein said composition when maintained at 100°C for 2 hours has a copper strip corrosion classification from about 1a to about 3b.

7. The composition of Claim 1, wherein said composition has a Falex reference wear of less than ten teeth.

8. The composition of Claim 1, wherein said composition has a Falex reference load of greater than about 4,500 pounds force.

9. The composition of Claim 1, wherein said composition has a Four-Ball wear scar diameter of less than about 0.07 mm.

10. The composition of Claim 1, wherein the metallic work pieces are nonferrous metallic work pieces.

11. A composition for lubricating nonferrous metallic work pieces comprising:

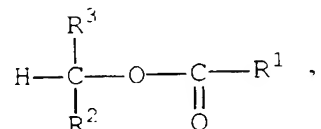
(a) an oil having a viscosity suitable for heavy duty metalworking operations; and

(b) sulfur being present in amounts of about 0.4 percent to about 12 percent by weight of said composition; wherein said composition does not corrode said nonferrous work piece.

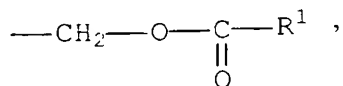
12. The composition of Claim 11, wherein said sulfur is not chemically bound to molecules in said oil.

13. The composition of Claim 11, wherein said composition when maintained at 100°C for 2 hours has a copper strip corrosion classification of about 1a to about 2a.

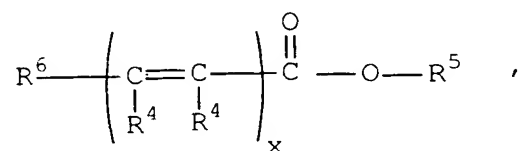
14. The composition of Claim 11, further comprising a fatty oil selected from the group consisting of a glyceride, an ester of a carboxylic acid, and combinations thereof, wherein said glyceride is represented by the formula of



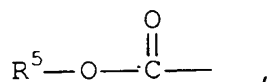
where  $\text{R}^1$  is a saturated or unsaturated  $\text{C}_3$  to  $\text{C}_{24}$  aliphatic hydrocarbon, and  $\text{R}^2$  or  $\text{R}^3$  is hydrogen or



wherein  $\text{R}^1$  is as defined above, and said ester is represented by the formula of



wherein  $\text{R}^4$  is hydrogen or a saturated or unsaturated  $\text{C}_3$  to  $\text{C}_{12}$  aliphatic hydrocarbon,  $x$  is 1, 2 or 3,  $\text{R}^5$  is a saturated or unsaturated  $\text{C}_3$  to  $\text{C}_{24}$  aliphatic hydrocarbon, and  $\text{R}^6$  is represented by the formula of



wherein  $\text{R}^5$  is as defined above, said fatty oil being present in an amount of about 5 to 30 volume percent based on the total composition and said fatty oil.

15. The composition of Claim 11, wherein said composition has a Falex reference wear of less than ten teeth.

16. The composition of Claim 11, wherein said composition has a Falex reference load of greater than about 4,500 pounds force.

17. The composition of Claim 11, wherein said composition has a Four-Ball wear scar diameter of less than about 0.07 mm.

18. The composition of Claim 11, wherein said composition has a viscosity of about 75 cSt to about 160 cSt at 25°C.

19. The composition of Claim 11, further comprising from about 0.0 to 4.0 weight percent chemically bound sulfur.

20. A method of making a composition which provides non-corrosive lubrication to metalworking processes comprising:

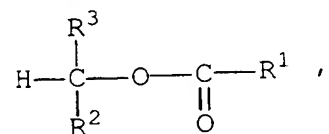
selecting a base oil having a viscosity of about 75 cSt to about 160 cSt at 25°C;

incorporating chemically unbound sulfur to said base oil to provide an extreme pressure lubricant; and

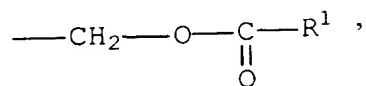
further incorporating a fatty oil to inhibit metal corrosion.

21. The method of Claim 20, wherein said composition has a Falex reference wear of less than ten teeth.

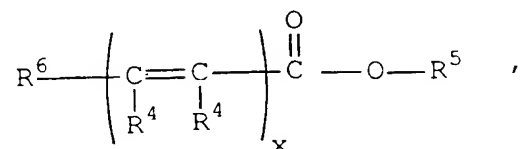
22. The method of Claim 20, wherein said fatty oil is selected from the group consisting of a glyceride, an ester of a carboxylic acid, and combinations thereof, wherein said glyceride is represented by the formula of



wherein R<sup>1</sup> is a saturated or unsaturated C<sub>3</sub> to C<sub>24</sub> aliphatic hydrocarbon and R<sup>2</sup> or R<sup>3</sup> is hydrogen or



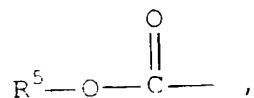
wherein R<sup>1</sup> is as defined above, and said ester is represented by the formula of



wherein R<sup>4</sup> is hydrogen or a saturated or unsaturated C<sub>3</sub> to C<sub>12</sub> aliphatic hydrocarbon, X is 1, 2 or 3, R<sup>5</sup> is a saturated or

- 23 -

unsaturated C<sub>3</sub> to C<sub>24</sub> aliphatic hydrocarbon, and R<sup>6</sup> is represented by the formula of



wherein R<sup>5</sup> is as defined above, and is combined into said composition in an amount from about 5 to about 30 volume percent based on the total composition and said fatty oil.

23. The method of Claim 20, further comprising incorporating from about 0.0 to 4.0 weight percent chemically bound sulfur.

24. A method of providing noncorrosive lubrication to the metalworking of nonferrous metal parts comprising:

providing a composition which includes a base oil having a viscosity of about 75 cSt to about 160 cSt at 25°C and free sulfur present in amounts sufficient to provide extreme pressure lubrication; and

applying said composition to the metal work piece and/or metal work tool during the metalworking process.



## INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US00/26167

## A. CLASSIFICATION OF SUBJECT MATTER

IPC(7) : C10M 125/06, 129/68

US CL : 508/152, 463, 486, 496; 72/42

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 508/152, 463, 486, 496; 72/42

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched  
NONEElectronic data base consulted during the international search (name of data base and, where practicable, search terms used)  
EAST search terms: metalworking, sulfur, free sulfur, glyceride

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 5,726,130 A (YAMANAKA) 10 March 1998 (10.03.1998), column 1, line 44 to column 2, line 14; column 3, lines 26-62 and column 5, lines 42-65.	1-24
Y	US 4,416,788 A (APIKOS) 22 November 1983 (22.11.1983), column 1, line 28 to column 2, line 45 and column 4, line 39 to column 6, line 24.	1-24
A	US 4,605,507 A (WINDGASSEN et al) 12 August 1986 (12.08.1986), column 2, line 19 to column 3, line 20.	1-24
A	US 4,125,471 A (VIENNA et al) 14 November 1978 (14.11.1978), column 3, line 7 to column 4, line 8.	1-24

☒ Further documents are listed in the continuation of Box C.
 ☐ See patent family annex.

* Special categories of cited documents	"T"	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"A" document defining the general state of the art which is not considered to be of particular relevance	"X"	document of particular relevance, the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"E" earlier document published on or after the international filing date	"Y"	document of particular relevance, the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"I" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"Δ"	document member of the same patent family
"O" document referring to an oral disclosure, use, exhibition or other means		
"P" document published prior to the international filing date but later than the priority date claimed		

Date of the actual completion of the international search

20 DECEMBER 2000

Date of mailing of the international search report

09 JAN 2001

Name and mailing address of the ISA/US  
Commissioner of Patents and Trademarks  
Box PCT  
Washington, D.C. 20231

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Authorized officer

ELLEN M. MCAVOY

Telephone No. (703) 308-0661

Jean Proctor  
Paralegal Specialist

## INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US00/26167

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 4,073,736 A (SCHICK et al) 14 February 1978 (14 02.1978), column 1, line 53 to column 4, line 20.	1-24

# PCT

## INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference LC-381-PCT	<b>FOR FURTHER ACTION</b> See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. PCT/US00/26167	International filing date (day month year) 22 September 2000 (22.09.2000)	Priority date (day month year) 22 September 1999 (22.09.1999)
International Patent Classification (IPC) or national classification and IPC IPC(7) C10M 125 (06), 129/68 and US Cl.: 508/152, 463, 486, 496, 72/42		
Applicant LOCTITE CORPORATION		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.
2. This REPORT consists of a total of 3 sheets, including this cover sheet.  
☒ This report is also accompanied by ANNEXES, i.e., sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of 4 sheets.

3. This report contains indications relating to the following items:
  - I ☒ Basis of the report
  - II ☐ Priority
  - III ☐ Non-establishment of report with regard to novelty, inventive step and industrial applicability
  - IV ☐ Lack of unity of invention
  - V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
  - VI ☐ Certain documents cited
  - VII ☐ Certain defects in the international application
  - VIII ☐ Certain observations on the international application

Date of submission of the demand 23 April 2001 (23.04.2001)	Date of completion of this report 10 December 2001 (10.12.2001)
Name and mailing address of the IPEA US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231 Facsimile No. (703)305-3230	Authorized officer Marian Knode Jean Procter Paralegal Telephone No. (703) 308-0661

## INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International Application No.

PCT/US00/26167

## I. Basis of the report

1 With regard to the elements of the international application \*

☐ the international application as originally filed.☒ the description:

pages 1-18, as originally filed

pages NONE, filed with the demand

pages NONE, filed with the letter of \_\_\_\_\_

☒ the claims:

pages 21, as originally filed

pages NONE, as amended (together with any statement) under Article 19

pages NONE, filed with the demand

pages 19, 20, 22, 23, filed with the letter of 01 October 2001 (01/10/2001)

☒ the drawings

pages none, as originally filed

pages NONE, filed with the demand

pages NONE, filed with the letter of \_\_\_\_\_

☐ the sequence listing part of the description:

pages NONE, as originally filed

pages NONE, filed with the demand

pages NONE, filed with the letter of \_\_\_\_\_

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language \_\_\_\_\_ which is:

☐ the language of a translation furnished for the purposes of international search (under Rule 23.1(b)).☐ the language of publication of the international application (under Rule 48.3(b)).☐ the language of the translation furnished for the purposes of international preliminary examination (under Rules 55.2 and/or 55.3).3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:☐ contained in the international application in printed form.☐ filed together with the international application in computer readable form.☐ furnished subsequently to this Authority in written form.☐ furnished subsequently to this Authority in computer readable form.☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished4. ☒ The amendments have resulted in the cancellation of☒ the description, pages none☒ the claims, Nos. 7 and 8☒ the drawings, sheets/fig none5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70 2(c)) \*\*

\* Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70 16 and 70 17).

\*\* Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.

**V. Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement****1. STATEMENT**

Novelty (N)	Claims <u>1-24</u>	YES
	Claims <u>NONE</u>	NO
Inventive Step (IS)	Claims <u>NONE</u>	YES
	Claims <u>1-24</u>	NO
Industrial Applicability (IA)	Claims <u>1-24</u>	YES
	Claims <u>NONE</u>	NO

**2. CITATIONS AND EXPLANATIONS**

Claims 1-24 lack an inventive step under PCT Article 33(3) as being obvious over Apikos (U.S. Patent No. 4,416,788) and Yamanaka (U.S. Patent No. 5,726,130), considered separately.

Apikos discloses a transparent lubricating oil composition useful as a high severity metal cutting oil comprising a major amount of oil of high lubricating viscosity of about 50 SUS to about 1000 SUS at 100 F and a minor amount of at least one sulfur-containing compound and an effective amount of elemental sulfur. See column 1, line 28 to column 2, line 45. The sulfur-containing compound may be any suitable compound soluble in lubricating oil at 40 F and which acts in combination with elemental sulfur to improve the extreme pressure properties of the composition. See column 2, line 46 to column 3, line 33. Elemental sulfur may be added to the composition in an amount of about 0.1 to about 1.5% by weight. This includes the amount of active sulfur added to the examples in the application of 1.4 weight % which results in a Falex Extreme Pressure Test value of 4500+ and a Falex Wear Test of zero. The elemental sulfur component of Apikos also acts to improve the extreme pressure properties of the composition. See column 3, lines 34-48. The composition may also comprise at least one ester component as a lubricity agent. See column 4, line 38 to column 6, line 20. Thus, Apikos clearly meets the limitations of the claims.

Yamanaka discloses a cutting or grinding oil composition comprising (A) a base oil such as a mineral oil having a kinematic viscosity in the range of 1.5 to 50 cSt at 40 C; (B) at least one compound selected from a dibasic acid having 2 to 6 carbon atoms, tribasic acids having 3-6 carbon atoms, and ester derivatives of these acids; and (C) a compound containing sulfur such as elemental sulfur. See column 1, line 45 to column 2, line 13. Elemental sulfur is present in the composition in the range of 0.05 to 2% by weight. The ester derivatives of the polybasic acids are set forth in column 3, lines 26-62, and elemental sulfur component (C) is set forth in column 5, lines 42-65. Thus, Yamanaka also clearly meets the limitations of the claims.

Claims 1-24 meet the criteria as defined under PCT Article 33(2) and 33(4).

----- NEW CITATIONS ----- ☐

What Is Claimed Is:

1. A composition for lubricating metallic work pieces comprising:

(a) an oil having a viscosity of about 75 cSt to about 160 cSt at 25°C;

(b) free sulfur in an amount sufficient to provide lubrication, and

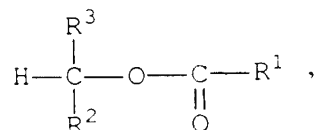
(c) a metal corrosion inhibitor to prevent corrosion of said work pieces;

wherein said lubrication is demonstrated by a Falex reference load of greater than about 4,500 pounds force and by a Falex reference wear of less than ten teeth.

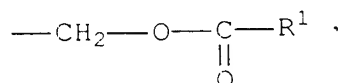
2. The composition of Claim 1, wherein said composition is a metalworking composition.

3. The composition of Claim 1, wherein said metal corrosion inhibitor is a fatty oil selected from the group consisting of a glyceride, an ester of a carboxylic acid, and combinations thereof,

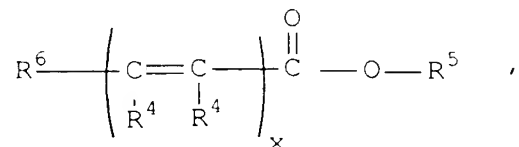
wherein said glyceride is represented by the formula of



wherein R<sup>1</sup> is a saturated or unsaturated C<sub>3</sub> to C<sub>24</sub> aliphatic hydrocarbon, and R<sup>2</sup> or R<sup>3</sup> is hydrogen or

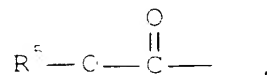


wherein R<sup>1</sup> is as defined above, and said ester is represented by the formula of



wherein R<sup>4</sup> is hydrogen or a saturated or unsaturated C<sub>3</sub> to C<sub>12</sub> aliphatic hydrocarbon, X is 1, 2 or 3, R<sup>5</sup> is a saturated or

unsaturated C<sub>3</sub> to C<sub>24</sub> aliphatic hydrocarbon, and R<sup>6</sup> is represented by the formula of



wherein R<sup>6</sup> is as defined above.

4. The composition of Claim 3, wherein said fatty oil is about 5 to about 30 volume percent based on said composition.

5. The composition of Claim 1, wherein said sulfur is present in amounts of from about 0.4 to about 12 percent by weight of said composition.

6. The composition of Claim 1, wherein said composition when maintained at 100°C for 2 hours has a copper strip corrosion classification from about 1a to about 3b.

9. The composition of Claim 1, wherein said composition has a Four-Ball wear scar diameter of less than about 0.07 mm.

10. The composition of Claim 1, wherein the metallic work pieces are nonferrous metallic work pieces.

11. A composition for lubricating nonferrous metallic work pieces comprising:

(a) an oil having a viscosity suitable for heavy duty metalworking operations; and

(b) free sulfur being present in amounts of about 0.4 percent to about 12 percent by weight of said composition;

wherein said composition does not corrode said nonferrous work pieces.

12. The composition of Claim 11, wherein said sulfur is not chemically bound to molecules in said oil.

13. The composition of Claim 11, wherein said composition when maintained at 100°C for 2 hours has a copper strip corrosion classification of about 1a to about 2a.

18. The composition of Claim 11, wherein said composition has a viscosity of about 75 cSt to about 160 cSt at 25°C.

19. The composition of Claim 11, further comprising from about 0.0 to 4.0 weight percent chemically bound sulfur.

20. A method of making a composition which provides non-corrosive lubrication to nonferrous metalworking processes comprising:

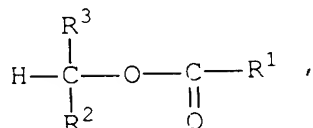
selecting a base oil having a viscosity of about 75 cSt to about 160 cSt at 25°C;

incorporating chemically unbound sulfur to said base oil to provide an extreme pressure lubricant, wherein the chemically unbound sulfur is incorporated in an amount from about 0.4 to about 12 weight percent of said composition; and

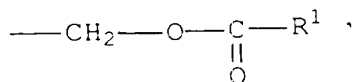
further incorporating a fatty oil to inhibit nonferrous metal corrosion.

21. The method of Claim 20, wherein said composition has a Falex reference wear of less than ten teeth.

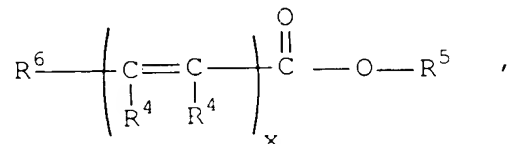
22. The method of Claim 20, wherein said fatty oil is selected from the group consisting of a glyceride, an ester of a carboxylic acid, and combinations thereof, wherein said glyceride is represented by the formula of



wherein R<sup>1</sup> is a saturated or unsaturated C<sub>3</sub> to C<sub>24</sub> aliphatic hydrocarbon and R<sup>2</sup> or R<sup>3</sup> is hydrogen or



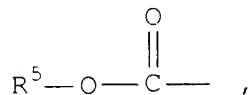
wherein R<sup>1</sup> is as defined above, and said ester is represented by the formula of



wherein R<sup>4</sup> is hydrogen or a saturated or unsaturated C<sub>3</sub> to C<sub>12</sub> aliphatic hydrocarbon, X is 1, 2 or 3, R<sup>5</sup> is a saturated or



unsaturated C<sub>3</sub> to C<sub>24</sub> aliphatic hydrocarbon, and R<sup>6</sup> is represented by the formula of



wherein R<sup>5</sup> is as defined above, and is combined into said composition in an amount from about 5 to about 30 volume percent based on the total composition and said fatty oil.

23. The method of Claim 20, further comprising incorporating from about 0.0 to 4.0 weight percent chemically bound sulfur.

24. A method of providing noncorrosive lubrication to the metalworking of a nonferrous metal part comprising:

providing a composition which includes a base oil having a viscosity of about 75 cSt to about 160 cSt at 25°C and free sulfur present in amounts sufficient to provide extreme pressure lubrication of a Falex reference load of greater than about 4,500 pounds force; and

applying said composition to the metal work part and/or a metal work tool during the metalworking process.

## PATENT COOPERATION TREATY

From the  
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

AUG 13 2001

PCT DEPARTMENT

To:  
STEVEN C. BAUMAN  
LOCTITE CORPORATION  
1001 TROUT BROOK CROSSING  
ROCKY HILL, CT 06067

WRITTEN OPINION

(PCT Rule 66)

Applicant's or agent's file reference LC-381-PCT		Date of Mailing (day/month/year) 09 AUG 2001
International application No. PCT/US00/26167		REPLY DUE within 2 months/days from the above date of mailing 10/09/01
International filing date (day/month/year) 22 September 2000 (22.09.2000)	Priority date (day/month/year) 22 September 1999 (22.09.1999)	
International Patent Classification (IPC) or both national classification and IPC IPC(7): C10M 125/06, 129/68 and US Cl.: 508/152, 463, 486, 496; 72/42		
Applicant LOCTITE CORPORATION		

- This written opinion is the first (first, etc.) drawn by this International Preliminary Examining Authority.
- This opinion contains indications relating to the following items:
  - ☒ Basis of the opinion
  - ☐ Priority
  - ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
  - ☐ Lack of unity of invention
  - ☒ Reasoned statement under Rule 66.2 (a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
  - ☐ Certain documents cited
  - ☐ Certain defects in the international application
  - ☐ Certain observations on the international application
- The applicant is hereby **invited to reply** to this opinion.
 

**When?** See the time limit indicated above. ~~The applicant may, before the expiration of that time limit, request this Authority to grant an extension. See rule 66.2(d).~~

**How?** By submitting a written reply, accompanied, where appropriate, by amendments, according to Rule 66.3. For the form and the language of the amendments, see Rules 66.8 and 66.9.

**Also** For an additional opportunity to submit amendments, see Rule 66.4.  
For the examiner's obligation to consider amendments and/or arguments, see Rule 66.4 bis.  
For an informal communication with the examiner, see Rule 66.6

**If no reply is filed**, the international preliminary examination report will be established on the basis of this opinion.
- The final date by which the international preliminary examination report must be established according to Rule 69.2 is: 22 January 2002 (22.01.2002).

Name and mailing address of the IPEA/US  
Commissioner of Patents and Trademarks  
Box PCT  
Washington, D.C. 20231  
Facsimile No. (703)305-3230

Authorized officer

Marian Knode

Telephone No. (703) 308-0661

Jean Proctor  
Paralegal Specialist

**WRITTEN OPINION**

Inter. application No. \_\_\_\_\_

PCT/US(00)/\_\_\_\_\_

**I. Basis of the opinion**

1. With regard to the elements of the international application:\*

- ☐ the international application as originally filed
- ☒ the description:
  - pages 1-18 \_\_\_\_\_, as originally filed
  - pages NONE \_\_\_\_\_, filed with the demand
  - pages NONE \_\_\_\_\_, filed with the letter of \_\_\_\_\_
- ☐ the claims:
  - pages 19-23 \_\_\_\_\_, as originally filed
  - pages NONE \_\_\_\_\_, as amended (together with any statement) under Article 19
  - pages NONE \_\_\_\_\_, filed with the demand
  - pages NONE \_\_\_\_\_, filed with the letter of \_\_\_\_\_
- ☒ the drawings:
  - pages none \_\_\_\_\_, as originally filed
  - pages NONE \_\_\_\_\_, filed with the demand
  - pages NONE \_\_\_\_\_, filed with the letter of \_\_\_\_\_
- ☐ the sequence listing part of the description:
  - pages NONE \_\_\_\_\_, as originally filed
  - pages NONE \_\_\_\_\_, filed with the demand
  - pages NONE \_\_\_\_\_, filed with the letter of \_\_\_\_\_

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.  
 These elements were available or furnished to this Authority in the following language \_\_\_\_\_ which is:

- ☐ the language of a translation furnished for the purposes of international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of the translation furnished for the purposes of international preliminary examination (under Rules 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the written opinion was drawn on the basis of the sequence listing:

- ☐ contained in the international application in printed form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. ☒ The amendments have resulted in the cancellation of:

- ☒ the description, pages none \_\_\_\_\_
- ☒ the claims, Nos. none \_\_\_\_\_
- ☒ the drawings, sheets/fig none \_\_\_\_\_

5. ☐ This opinion has been drawn as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).

\* Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this opinion as "originally filed."

**V. Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**

**1. STATEMENT**

Novelty (N)	Claims <u>1-24</u>	YES
	Claims <u>NONE</u>	NO
Inventive Step (IS)	Claims <u>NONE</u>	YES
	Claims <u>1-24</u>	NO
Industrial Applicability (IA)	Claims <u>1-24</u>	YES
	Claims <u>NONE</u>	NO

**2. CITATIONS AND EXPLANATIONS**

Claims 1-24 lack an inventive step under PCT Article 33(3) as being obvious over Apikos (U.S. Patent No. 4,416,788) and Yamanaka (U.S. Patent No. 5,726,130), considered separately.

Apikos discloses a transparent lubricating oil composition useful as a high severity metal cutting oil comprising a major amount of oil of lubricating viscosity and a minor amount of at least one sulfur-containing compound and an effective amount of elemental sulfur. See column 1, line 28 to column 2, line 45. The sulfur-containing compound may be any suitable compound soluble in lubricating oil at 40 F and which acts in combination with elemental sulfur to improve the extreme pressure properties of the composition. See column 2, line 46 to column 3, line 33. Elemental sulfur may be added to the composition in an amount of about 0.1 to about 1.5% by weight. See column 3, lines 34-48. The composition may also comprise at least one ester component as a lubricity agent. See column 4, line 38 to column 6, line 20. Thus, Apikos clearly meets the limitations of the claims.

Yamanaka discloses a cutting or grinding oil composition comprising (A) a base oil such as a mineral oil having a kinematic viscosity in the range of 1.5 to 50 cSt at 40 C; (B) at least one compound selected from a dibasic acid having 2 to 6 carbon atoms, tribasic acids having 3-6 carbon atoms, and ester derivatives of these acids; and (C) a compound containing sulfur such as elemental sulfur. See column 1, line 45 to column 2, line 13. The ester derivatives of the polybasic acids are set forth in column 3, lines 26-62, and elemental sulfur component (C) is set forth in column 5, lines 42-65. Thus, Yamanaka also clearly meets the limitations of the claims.

----- NEW CITATIONS -----

WRITTEN OPINION

International application No.  
PCT/US/01/067

**Supplemental Box**

(To be used when the space in any of the preceding boxes is not sufficient)

**TIME LIMIT:**

The time limit set for response to a Written Opinion may not be extended. 37 CFR 1.484(d). Any response received after the expiration of the time limit set in the Written Opinion will not be considered in preparing the International Preliminary Examination Report.

## PCT REQUEST

Original (for SUBMISSION) - printed on 22.09.2000 05:37:18 PM

0	<b>For receiving Office use only</b>	
0-1	International Application No.	
0-2	International Filing Date	
0-3	Name of receiving Office and "PCT International Application"	
0-4	<b>Form - PCT/RO/101 PCT Request</b>	
0-4-1	Prepared using	PCT-EASY Version 2.91 (updated 01.07.2000)
0-5	<b>Petition</b> The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty	
0-6	Receiving Office (specified by the applicant)	United States Patent and Trademark Office (USPTO) (RO/US)
0-7	Applicant's or agent's file reference	LC-381-PCT
I	Title of invention	NON-STAINING, ACTIVE METAL-WORKING FLUID
II	<b>Applicant</b>	
II-1	This person is:	applicant only
II-2	Applicant for	all designated States except US
II-4	Name	LOCTITE CORPORATION
II-5	Address:	1001 Trout Brook Crossing Rocky Hill, CT 06067 United States of America
II-6	State of nationality	US
II-7	State of residence	US
II-8	Telephone No.	(860) 571-5100
II-9	Facsimile No.	(860) 571-5465
II-10	e-mail	steve.bauman@loctite.com
III-1	<b>Applicant and/or inventor</b>	
III-1-1	This person is:	applicant and inventor
III-1-2	Applicant for	US only
III-1-4	Name (LAST, First)	FISHER, Edward, A.Y.
III-1-5	Address:	167 New Britain Avenue Rocky Hill, CT 06067 United States of America
III-1-6	State of nationality	US
III-1-7	State of residence	US

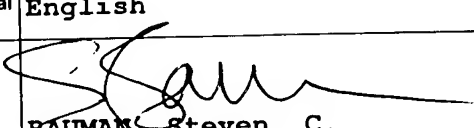
## PCT REQUEST

Original (for SUBMISSION) - printed on 22.09.2000 05:37:18 PM

IV-1	<b>Agent or common representative; or address for correspondence</b> The person identified below is hereby/has been appointed to act on behalf of the applicant(s) before the competent International Authorities as:	<b>agent</b>
IV-1-1	Name (LAST, First)	BAUMAN, Steven, C.
IV-1-2	Address:	LOCTITE CORPORATION 1001 Trout Brook Crossing Rocky Hill, CT 06067 United States of America
IV-1-3	Telephone No.	(860) 571-5001
IV-1-4	Facsimile No.	(860) 571-5028
IV-1-5	e-mail	steve.bauman@loctite.com
V	<b>Designation of States</b>	
V-1	Regional Patent (other kinds of protection or treatment, if any, are specified between parentheses after the designation(s) concerned)	AP: GH GM KE LS MW MZ SD SL SZ TZ UG ZW and any other State which is a Contracting State of the Harare Protocol and of the PCT EA: AM AZ BY KG KZ MD RU TJ TM and any other State which is a Contracting State of the Eurasian Patent Convention and of the PCT EP: AT BE CH&LI CY DE DK ES FI FR GB GR IE IT LU MC NL PT SE and any other State which is a Contracting State of the European Patent Convention and of the PCT OA: BF BJ CF CG CI CM GA GN GW ML MR NE SN TD TG and any other State which is a member State of OAPI and a Contracting State of the PCT
V-2	National Patent (other kinds of protection or treatment, if any, are specified between parentheses after the designation(s) concerned)	AE AG AL AM AT AU AZ BA BB BG BR BY BZ CA CH&LI CN CR CU CZ DE DK DM DZ EE ES FI GB GD GE GH GM HR HU ID IL IN IS JP KE KG KP KR KZ LC LK LR LS LT LU LV MA MD MG MK MN MW MX MZ NO NZ PL PT RO RU SD SE SG SI SK SL TJ TM TR TT TZ UA UG US UZ VN YU ZA ZW
V-5	<b>Precautionary Designation Statement</b> In addition to the designations made under items V-1, V-2 and V-3, the applicant also makes under Rule 4.9(b) all designations which would be permitted under the PCT except any designation(s) of the State(s) indicated under item V-6 below. The applicant declares that those additional designations are subject to confirmation and that any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit.	

## PCT REQUEST

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V-6	Exclusion(s) from precautionary designations	NONE	
VI-1	Priority claim of earlier national application		
VI-1-1	Filing date	22 September 1999 (22.09.1999)	
VI-1-2	Number	60/155,345	
VI-1-3	Country	US	
VII-1	International Searching Authority Chosen	United States Patent and Trademark Office (USPTO) (ISA/US)	
VII-2	Request to use results of earlier search; reference to that search		
VII-2-1	Date		
VII-2-2	Number		
VII-2-3	Country (or regional Office)	US	
VIII	Check list	number of sheets	electronic file(s) attached
VIII-1	Request	4	-
VIII-2	Description	18	-
VIII-3	Claims	5	-
VIII-4	Abstract	1	lc-381-pct.txt
VIII-5	Drawings	0	-
VIII-7	TOTAL	28	
	Accompanying items	paper document(s) attached	electronic file(s) attached
VIII-8	Fee calculation sheet	✓	-
VIII-16	PCT-EASY diskette	-	diskette
VIII-17	Other (specified):	Return Receipt Postcard	-
VIII-18	Figure of the drawings which should accompany the abstract		
VIII-19	Language of filing of the international application	English	
IX-1	Signature of applicant or agent	 Counsel, Intellectual Property	
IX-1-1	Name (LAST, First)	BAUMAN, Steven, C.	

## FOR RECEIVING OFFICE USE ONLY

10-1	Date of actual receipt of the purported international application	
10-2	Drawings:	
10-2-1	Received	
10-2-2	Not received	
10-3	Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application	
10-4	Date of timely receipt of the required corrections under PCT Article 11(2)	
10-5	International Searching Authority	ISA/US
10-6	Transmittal of search copy delayed until search fee is paid	



## PCT REQUEST

Original (for SUBMISSION) - printed on 22.09.2000 05:37:18 PM

## FOR INTERNATIONAL BUREAU USE ONLY

11-1	Date of receipt of the record copy by the International Bureau	
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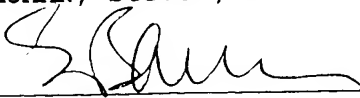
**PCT (ANNEX - FEE CALCULATION SHEET)**Original (for **SUBMISSION**) - printed on 22 09 2000 05:37:18 PM

(This sheet is not part of and does not count as a sheet of the international application)

0	For receiving Office use only		
0-1	International Application No.		
0-2	Date stamp of the receiving Office		
0-4	Form - PCT/RO/101 (Annex)		
0-4-1	PCT Fee Calculation Sheet Prepared using	PCT-EASY Version 2.91 (updated 01.07.2000)	
0-9	Applicant's or agent's file reference	LC-381-PCT	
2	Applicant	LOCTITE CORPORATION, et al.	
12	Calculation of prescribed fees	fee amount/multiplier	total amounts (USD)
12-1	Transmittal fee T	⇒	240
12-2	Search fee S	⇒	700
12-3	International fee		
	Basic fee (first 30 sheets) b1	427	
12-4	Remaining sheets	0	
12-5	Additional amount (X) 10		
12-6	Total additional amount b2	0	
12-7	b1 + b2 = B	427	
12-8	Designation fees		
	Number of designations contained in international application	87	
12-9	Number of designation fees payable (maximum 8)	8	
12-10	Amount of designation fee (X) 92		
12-11	Total designation fees D	736	
12-12	PCT-EASY fee reduction R	-132	
12-13	Total International fee (B+D-R) I	⇒	1,031
12-17	TOTAL FEES PAYABLE (T+S+I+P)	⇒	1,971
12-19	Mode of payment	authorization to charge deposit account	
12-20	Deposit account instructions The receiving Office:	United States Patent and Trademark Office (USPTO) (RO/US)	
12-20-1	is hereby authorized to charge the total fees indicated above to my deposit account	✓	
12-20-2	is hereby authorized to charge any deficiency or credit any over-payment in the total fees indicated above to my deposit account	✓	
12-20-3	is hereby authorized to charge the fee for preparation and transmittal of the priority document to the International Bureau of WIPO to my deposit account	✓	
12-21	Deposit account No.	12-2135	
12-22	Date	22 September 2000 (22.09.2000)	

## PCT (ANNEX - FEE CALCULATION SHEET)

Original (for SUBMISSION) - printed on 22 09 2000 05:37:18 PM

12-23	Name and signature	BAUMAN, Steven, C. 
-------	--------------------	--

## VALIDATION LOG AND REMARKS

13-2-6	Validation messages Contents	Yellow! The power of attorney or a copy of the general power of attorney will need to be furnished unless all applicants sign the request form.
		Green? The international application contains no drawings. Please verify.
		Green? Priority 1. The priority document is not enclosed. (The applicant must furnish it within 16 months from the earliest priority date claimed)
13-2-7	Validation messages Fees	Green? Please confirm that fee schedule utilized is the latest available
13-2-8	Validation messages Payment	Green? Please ensure that you have a valid deposit account with the receiving Office selected.

The demand must be filed directly with the competent International Examining Authority or, if two or more authorities are competent, with the one chosen by the applicant. The full name and two-letter code of that Authority may be indicated by the applicant on the line below  
 IPEA/ US

# PCT

## CHAPTER II

### DEMAND

Under Article 31 of the Patent Cooperation Treaty:  
 The undersigned requests that the international application specified below be the subject of  
 International preliminary examination according to the Patent Cooperation Treaty and  
 hereby elects all eligible States (except where otherwise indicated).

For International Preliminary Examining Authority use only		
Identification of IPEA		Date of receipt of DEMAND
<b>Box No. I IDENTIFICATION OF THE INTERNATIONAL APPLICATION</b>		Applicant's or agent's file reference <b>LC-381 PCT</b>
International application No. <b>PCT/US00/26167</b>	International filing date (day/month/year) <b>22 SEPTEMBER 2000 (22.09.00)</b>	(Earliest) Priority date (day/month/year) <b>22 SEPTEMBER 1999 (22.09.99)</b>
Title of invention <b>NON-STAINING, ACTIVE METAL-WORKING FLUID</b>		
<b>Box No. II APPLICANT(S)</b>		
Name and address: (Family name followed by given name; for a legal entity, full official designation The address must include postal code and name of country.) <b>LOCTITE CORPORATION 1001 Trout Brook Crossing Rocky Hill, Connecticut 06067 US</b>		Telephone No.: <b>860.571.5000</b>
		Facsimile No.: <b>860.571.5465</b>
		Teleprinter No.:
State (that is, country) of nationality: <b>US</b>	State (that is, country) of residence: <b>US</b>	
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.) <b>FISHER, Edward A.Y. 9 Barbara Avenue East Hampton, Connecticut 06424 US</b>		
State (that is, country) of nationality: <b>US</b>	State (that is, country) of residence: <b>US</b>	
Name and address: (Family name followed by given name, for a legal entity, full official designation. The address must include postal code and name of country.)		
State (that is, country) of nationality:		
State (that is, country) of residence:		
<input type="checkbox"/> Further applicants are indicated on a continuation sheet.		

Form PCT/IPEA/401 (first sheet) (July 1998; reprint July 1999)

See Notes to the demand form

**Box No. III AGENT OR COMMON REPRESENTATIVE; OR ADDRESS FOR CORRESPONDENCE**The following person is ☒ agent ☐ common representativeand ☒ has been appointed earlier and represents the applicant(s) also for international preliminary examination☐ is hereby appointed and any earlier appointment of (an) agent(s)/common representative is hereby revoked.☐ is hereby appointed, specifically for the procedure before the International Preliminary Examining Authority, in addition to the agent(s)/common representative appointed earlier.Name and address: *(Family name followed by given name, for a legal entity, full official designation  
The address must include postal code and name of country)*

BAUMAN, Steven C.  
LOCTITE CORPORATION  
1001 Trout Brook Crossing  
Rocky Hill, Connecticut 06067  
US

Telephone No.:

860.571-5001

Facsimile No.:

860.571.5028

Teleprinter No.:

☐ Address for correspondence: Mark this check-box where no agent or common representative is/has been appointed and the space above is used instead to indicate a special address to which correspondence should be sent.**Box No. IIV BASIS FOR INTERNATIONAL PRELIMINARY EXAMINATION****Statement concerning amendments:\***

1. The applicant wishes the international preliminary examination to start on the basis of:

☒ the international application as originally filed

the description

☒

as originally filed

☐

as amended under Article 34

the claims

☒

as originally filed

☐

as amended under Article 19 (together with any accompanying statement)

☐

as amended under Article 34

the drawings

☐

as originally filed

☐

as amended under Article 34

2. ☐ The applicant wishes any amendment to the claims under Article 19 to be considered reversed.3. ☐ The applicant wishes the start of the international preliminary examination to be postponed until the expiration of 20 months from the priority date unless the International Preliminary Examining Authority receives a copy of any amendments made under Article 19 or a notice from the applicant that he does not wish to make such amendments (Rule 69.1(d)). *(This check-box may be marked only where the time limit under Article 19 has not yet expired.)*

\* Where no check-box is marked, international preliminary examination will start on the basis of the international application as originally filed or, where a copy of amendments to the claims under Article 19 and/or amendments of the international application under Article 34 are received by the International Preliminary Examining Authority before it has begun to draw up a written opinion or the international preliminary examination report, as so amended.

Language for the purposes of international preliminary examination: .....English.....

☒

which is the language in which the international application was filed.

☐

which is the language of a translation furnished for the purposes of international search.

☐

which is the language of publication of the international application.

☐

which is the language of the translation (to be) furnished for the purposes of international preliminary examination.

**Box No. V ELECTION OF STATES**

The applicant hereby elects all eligible States *(that is, all States which have been designated and which are bound by Chapter II of the PCT)*  
excluding the following States which the applicant wishes not to elect:

## Box No. VI CHECK LIST

The demand is accompanied by the following elements, in the language referred to in Box No. IV, for the purposes of international preliminary examination

- |   |   |        |
|---|---|--------|
| 1 | translation of international application                              | sheets |
| 2 | amendments under Article 34   | sheets |
| 3 | copy (or, where required, translation) of amendments under Article 19 | sheets |
| 4 | copy (or, where required, translation) of statement under Article 19  | sheets |
| 5 | letter  | sheets |
| 6 | other (specify)   | sheets |

For International Preliminary Examining Authority use only

received	not received
----------	--------------

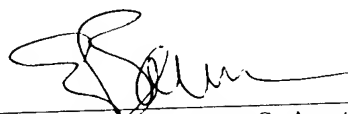
<input type="checkbox"/>	<input type="checkbox"/>
<input type="checkbox"/>	<input type="checkbox"/>
<input type="checkbox"/>	<input type="checkbox"/>
<input type="checkbox"/>	<input type="checkbox"/>
<input type="checkbox"/>	<input type="checkbox"/>
<input type="checkbox"/>	<input type="checkbox"/>

The demand is also accompanied by the item(s) marked below:

- |  |   |
|--|---|
| 1. <input checked="" type="checkbox"/> fee calculation sheet                             | 4. <input type="checkbox"/> statement explaining lack of signature                                  |
| 2. <input type="checkbox"/> separate signed power of attorney                            | 5. <input type="checkbox"/> nucleotide and or amino acid sequence listing in computer readable form |
| 3. <input type="checkbox"/> copy of general power of attorney; reference number, if any: | 6. <input checked="" type="checkbox"/> other (specify): RETURN RECEIPT POSTCARD                     |

## Box No. VII SIGNATURE OF APPLICANT, AGENT OR COMMON REPRESENTATIVE

Next to each signature, indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the demand)



BAUMAN, Steven C., Agent  
(Registration No. 33,832)

For International Preliminary Examining Authority use only

1. Date of actual receipt of DEMAND:

2. Adjusted date of receipt of demand due to CORRECTIONS under Rule 60.1(b):

3. ☐ The date of receipt of the demand is AFTER the expiration of 19 months from the priority date and item 4 or 5, below, does not apply. ☐ The applicant has been informed accordingly.

4. ☐ The date of receipt of the demand is WITHIN the period of 19 months from the priority date as extended by virtue of Rule 80.5.

5. ☐ Although the date of receipt of the demand is after the expiration of 19 months from the priority date, the delay in arrival is EXCUSED pursuant to Rule 82.

For International Bureau use only

Demand received from IPEA on:

## FEE CALCULATION SHEET

Annex to the Demand for international preliminary examination

<table border="1" style="width: 100%; border-collapse: collapse;"><tr><td style="width: 50%; padding: 2px;">International application No.</td><td style="width: 50%; padding: 2px;">PCT/US00/26167</td></tr><tr><td style="padding: 2px;">Applicant's or agent's file reference</td><td style="padding: 2px;">LC-381 PCT</td></tr></table> <p style="margin-top: 5px;">Applicant <b>LOCTITE CORPORATION</b> <b>FISHER, Edward A.Y.</b></p> <p>Calculation of prescribed fees</p> <table style="width: 100%;"><tr><td style="width: 60%;">1. Preliminary examination fee .....</td><td style="width: 20%; text-align: center;">490.00</td><td style="width: 20%; text-align: center;"><input type="checkbox"/> P</td></tr><tr><td colspan="3"> </td></tr><tr><td>2. Handling fee (<i>Applicants from certain States are entitled to a reduction of 75% of the handling fee. Where the applicant is (or all applicants are) so entitled, the amount to be entered at H is 25% of the handling fee.</i>) .....</td><td style="text-align: center;">153.00</td><td style="text-align: center;"><input type="checkbox"/> H</td></tr><tr><td colspan="3"> </td></tr><tr><td>3. Total of prescribed fees Add the amounts entered at P and H and enter total in the TOTAL box .....</td><td style="text-align: center;">643.00</td><td></td></tr><tr><td></td><td style="text-align: center;"><b>TOTAL</b></td><td></td></tr></table>	International application No.	PCT/US00/26167	Applicant's or agent's file reference	LC-381 PCT	1. Preliminary examination fee .....	490.00	<input type="checkbox"/> P				2. Handling fee ( <i>Applicants from certain States are entitled to a reduction of 75% of the handling fee. Where the applicant is (or all applicants are) so entitled, the amount to be entered at H is 25% of the handling fee.</i> ) .....	153.00	<input type="checkbox"/> H				3. Total of prescribed fees Add the amounts entered at P and H and enter total in the TOTAL box .....	643.00			<b>TOTAL</b>		<div style="border: 1px solid black; padding: 2px; margin-bottom: 10px;">For International Preliminary Examining Authority use only</div> <div style="border: 1px solid black; padding: 2px; margin-bottom: 10px;">Date stamp of the IPEA</div> <div style="border: 1px solid black; height: 200px; margin-bottom: 10px;"></div> <div style="border: 1px solid black; height: 100px;"></div>
International application No.	PCT/US00/26167																						
Applicant's or agent's file reference	LC-381 PCT																						
1. Preliminary examination fee .....	490.00	<input type="checkbox"/> P																					
2. Handling fee ( <i>Applicants from certain States are entitled to a reduction of 75% of the handling fee. Where the applicant is (or all applicants are) so entitled, the amount to be entered at H is 25% of the handling fee.</i> ) .....	153.00	<input type="checkbox"/> H																					
3. Total of prescribed fees Add the amounts entered at P and H and enter total in the TOTAL box .....	643.00																						
	<b>TOTAL</b>																						

**Mode of Payment**

<input checked="" type="checkbox"/> authorization to charge deposit account with the IPEA (see below)	<input type="checkbox"/> Cash
<input type="checkbox"/> Cheque	<input type="checkbox"/> Revenue stamps
<input type="checkbox"/> postal money order	<input type="checkbox"/> Coupons
<input type="checkbox"/> bank draft	<input type="checkbox"/> Other ( <i>specify</i> ):

Deposit Account Authorization (*this mode of payment may not be available at all IPEAs*)

The IPEA/ US ☒ is hereby authorized to charge the total fees indicated above to my deposit account.


☒ (*this check-box may be marked only if the conditions for deposit accounts of the IPEA so permit*) is hereby authorized to charge any deficiency or credit any overpayment in the total fees indicated above to my deposit account.

12-2135

Deposit Account Number

20 April 2000

Date (day/month/year)



Signature      Steven C. Bauman

# PATENT COOPERATION TREATY

From the  
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To:  
STEVEN C. BAUMAN  
LOCTITE CORPORATION  
1001 TROUT BROOK CROSSING  
ROCKY HILL, CT 06067

## PCT

### NOTIFICATION OF TRANSMITTAL OF INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Rule 71.1)

Date of Mailing  
(day/month/year)

03 JAN 2002

Applicant's or agent's file reference

LC-381 PCT

#### IMPORTANT NOTIFICATION

International application No.

PCT/US00/26167

International filing date (day/month/year)

22 September 2000 (22.09.2000)

Priority date (day/month/year)

22 September 1999 (22.09.1999)

Applicant

LOCTITE CORPORATION

1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the international preliminary examination report and its annexes, if any, established on the international application.
2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.
3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.
4. **REMINDER**

The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices)(Article 39(1))(see also the reminder sent by the International Bureau with Form PCT/IB/301).

Where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary examination report. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the PCT Applicant's Guide.

Name and mailing address of the IPEA/US

Commissioner of Patents and Trademarks

Box PCT

Washington, D.C. 20231

Facsimile No. (703)305-3230

Form PCT/IPEA/416 (July 1992)

Authorized officer

Marian Knode

Jean Proctor  
Paralegal Services Unit

Telephone No. (703) 308-0661



# PCT

## INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference LC 381-PCT	<b>FOR FURTHER ACTION</b> See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No PCT/US00/26167	International filing date (day/month/year) 22 September 2000 (22 09 2000)	Priority date (day/month/year) 22 September 1999 (22 09 1999)
International Patent Classification (IPC) or national classification and IPC IPC(7): C10M 125/06, 129/68 and US Cl.: 508/152, 463, 486, 496, 72/42		
Applicant LOCTITE CORPORATION		
<p>1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.</p> <p>2. This REPORT consists of a total of <u>3</u> sheets, including this cover sheet.</p> <p><input checked="" type="checkbox"/> This report is also accompanied by ANNEXES, i.e., sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).</p> <p>These annexes consist of a total of <u>4</u> sheets.</p>		
<p>3. This report contains indications relating to the following items:</p> <p>I <input checked="" type="checkbox"/> Basis of the report</p> <p>II <input type="checkbox"/> Priority</p> <p>III <input type="checkbox"/> Non-establishment of report with regard to novelty, inventive step and industrial applicability</p> <p>IV <input type="checkbox"/> Lack of unity of invention</p> <p>V <input checked="" type="checkbox"/> Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement</p> <p>VI <input type="checkbox"/> Certain documents cited</p> <p>VII <input type="checkbox"/> Certain defects in the international application</p> <p>VIII <input type="checkbox"/> Certain observations on the international application</p>		
Date of submission of the demand 23 April 2001 (23 04 2001)	Date of completion of this report 10 December 2001 (10 12 2001)	
Name and mailing address of the IPEA/US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231 Facsimile No. (703)305-3230	Authorized officer Marian Knode Jean Procter Paralegal S. J. J. J. J. Telephone No. (703) 308-0661	

## 1. Basis of the report

1. With regard to the elements of the international application:\*

☐ the international application as originally filed.☒ the description:

pages 1-18 \_\_\_\_\_ as originally filed

pages NONE \_\_\_\_\_, filed with the demandpages NONE \_\_\_\_\_, filed with the letter of \_\_\_\_\_☒ the claims:

pages 21 \_\_\_\_\_, as originally filed

pages NONE \_\_\_\_\_, as amended (together with any statement) under Article 19pages NONE \_\_\_\_\_, filed with the demandpages 19, 20, 22, 23 \_\_\_\_\_, filed with the letter of 01 October 2001 (01.10.2001)☒ the drawingspages none \_\_\_\_\_, as originally filedpages NONE \_\_\_\_\_, filed with the demandpages NONE \_\_\_\_\_, filed with the letter of \_\_\_\_\_☐ the sequence listing part of the description:pages NONE \_\_\_\_\_, as originally filedpages NONE \_\_\_\_\_, filed with the demandpages NONE \_\_\_\_\_, filed with the letter of \_\_\_\_\_2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language \_\_\_\_\_ which is:

☐ the language of a translation furnished for the purposes of international search (under Rule 23.1(b)).☐ the language of publication of the international application (under Rule 48.3(b)).☐ the language of the translation furnished for the purposes of international preliminary examination (under Rules 55.2 and/or 55.3).3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:☐ contained in the international application in printed form.☐ filed together with the international application in computer readable form.☐ furnished subsequently to this Authority in written form.☐ furnished subsequently to this Authority in computer readable form.☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.4. ☒ The amendments have resulted in the cancellation of:☒ the description, pages none☒ the claims, Nos. 7 and 8☒ the drawings, sheets/fig none5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)) \*\*

\* Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17).

\*\* Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.

# INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International Application No.  
PCT/US00/20167

## V. Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

### 1. STATEMENT

Novelty (N)	Claims <u>1-24</u>	YES
	Claims <u>NONE</u>	NO
Inventive Step (IS)	Claims <u>NONE</u>	YES
	Claims <u>1-24</u>	NO
Industrial Applicability (IA)	Claims <u>1-24</u>	YES
	Claims <u>NONE</u>	NO

### 2. CITATIONS AND EXPLANATIONS

Claims 1-24 lack an inventive step under PCT Article 33(3) as being obvious over Apikos (U.S. Patent No. 4,416,788) and Yamanaka (U.S. Patent No. 5,726,130), considered separately.

Apikos discloses a transparent lubricating oil composition useful as a high severity metal cutting oil comprising a major amount of oil of high lubricating viscosity of about 50 SUS to about 1000 SUS at 100 F and a minor amount of at least one sulfur-containing compound and an effective amount of elemental sulfur. See column 1, line 28 to column 2, line 45. The sulfur-containing compound may be any suitable compound soluble in lubricating oil at 40 F and which acts in combination with elemental sulfur to improve the extreme pressure properties of the composition. See column 2, line 46 to column 3, line 33. Elemental sulfur may be added to the composition in an amount of about 0.1 to about 1.5% by weight. This includes the amount of active sulfur added to the examples in the application of 1.4 weight % which results in a Falex Extreme Pressure Test value of 4500+ and a Falex Wear Test of zero. The elemental sulfur component of Apikos also acts to improve the extreme pressure properties of the composition. See column 3, lines 34-48. The composition may also comprise at least one ester component as a lubricity agent. See column 4, line 38 to column 6, line 20. Thus, Apikos clearly meets the limitations of the claims.

Yamanaka discloses a cutting or grinding oil composition comprising (A) a base oil such as a mineral oil having a kinematic viscosity in the range of 1.5 to 50 cSt at 40 C; (B) at least one compound selected from a dibasic acid having 2 to 6 carbon atoms, tribasic acids having 3-6 carbon atoms, and ester derivatives of these acids; and (C) a compound containing sulfur such as elemental sulfur. See column 1, line 45 to column 2, line 13. Elemental sulfur is present in the composition in the range of 0.05 to 2% by weight. The ester derivatives of the polybasic acids are set forth in column 3, lines 26-62, and elemental sulfur component (C) is set forth in column 5, lines 42-65. Thus, Yamanaka also clearly meets the limitations of the claims.

Claims 1-24 meet the criteria as defined under PCT Article 33(2) and 33(4).

----- NEW CITATIONS ----- ☐

JAN 12 2001

From the INTERNATIONAL SEARCHING AUTHORITY

To: STEVEN C. BAUMAN  
LOCTITE CORPORATION  
1001 TROUT BROOK CROSSING  
ROCKY HILL, CT 06067

RD  
1/22/01

PCT

NOTIFICATION OF TRANSMITTAL OF  
THE INTERNATIONAL SEARCH REPORT  
OR THE DECLARATION

(PCT Rule 44.1)

Date of Mailing (day/month/year)	09 JAN 2001
Applicant's or agent's file reference LC-381-PCT	FOR FURTHER ACTION See paragraphs 1 and 4 below
International application No. PCT/US00/26167	International filing date (day/month/year) 22 SEPTEMBER 2000
Applicant LOCTITE CORPORATION	

1. ☒ The applicant is hereby notified that the international search report has been established and is transmitted herewith.
- Filing of amendments and statement under Article 19:**  
The applicant is entitled, if he so wishes, to amend the claims of the international application (see Rule 46):
- When?** The time limit for filing such amendments is normally 2 months from the date of transmittal of the international search report; however, for more details, see the notes on the accompanying sheet.
- Where?** Directly to the International Bureau of WIPO  
34, chemin des Colombettes  
1211 Geneva 20, Switzerland  
Facsimile No.: (41-22) 740.14.35
- For more detailed instructions, see the notes on the accompanying sheet.
2. ☐ The applicant is hereby notified that no international search report will be established and that the declaration under Article 17(2)(a) to that effect is transmitted herewith.
3. ☐ With regard to the protest against payment of (an) additional fee(s) under Rule 40.2, the applicant is notified that:
- ☐ the protest together with the decision thereon has been transmitted to the International Bureau together with the applicant's request to forward the texts of both the protest and the decision thereon to the designated Offices.
- ☐ no decision has been made yet on the protest; the applicant will be notified as soon as a decision is made.
4. **Further action(s):** The applicant is reminded of the following:
- Shortly after **18 months** from the priority date, the international application will be published by the International Bureau. If the applicant wishes to avoid or postpone publication, a notice of withdrawal of the international application, or of the priority claim, must reach the International Bureau as provided in rules 90 *bis* 1 and 90 *bis* 3, respectively, before the completion of the technical preparations for international publication.
- Within **19 months** from the priority date, a demand for international preliminary examination must be filed if the applicant wishes to postpone the entry into the national phase until 30 months from the priority date (in some Offices even later).
- Within **20 months** from the priority date, the applicant must perform the prescribed acts for entry into the national phase before all designated Offices which have not been elected in the demand or in a later election within 19 months from the priority date or could not be elected because they are not bound by Chapter II.

Name and mailing address of the ISA/US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231 Facsimile No. (703) 305-3230	Authorized officer ELLEN M. MCAVOY Jean Proctor Paraegal Specialist Telephone No. (703) 308-0601
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# PATENT COOPERATION TREATY

## PCT

### INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference LC-381-PCT	<div style="display: flex; justify-content: space-between;"> <div style="text-align: center;">FOR FURTHER ACTION</div> <div>see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.</div> </div>
International application No. PCT/US00/26167	<div style="display: flex; justify-content: space-between;"> <div>International filing date (day/month/year) 22 SEPTEMBER 2000</div> <div>(Earliest) Priority Date (day/month/year) 22 SEPTEMBER 1999</div> </div>
Applicant LOCTITE CORPORATION	

This international search report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This international search report consists of a total of 3 sheets.

☒ It is also accompanied by a copy of each prior art document cited in this report.

#### 1. Basis of the report

a. With regard to the **language**, the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.

☐ the international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).

b. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international search was carried out on the basis of the sequence listing:

☐ contained in the international application in written form.

☐ filed together with the international application in computer readable form.

☐ furnished subsequently to this Authority in written form.

☐ furnished subsequently to this Authority in computer readable form.

☐ the statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.

☐ the statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

2. ☐ Certain claims were found unsearchable (See Box I).

3. ☐ Unity of invention is lacking (See Box II).

4. With regard to the **title**,

☒ the text is approved as submitted by the applicant.

☐ the text has been established by this Authority to read as follows:

5. With regard to the **abstract**,

☒ the text is approved as submitted by the applicant.

☐ the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.

6. The figure of the **drawings** to be published with the abstract is Figure No. \_\_\_\_\_

☐ as suggested by the applicant.

☐ because the applicant failed to suggest a figure.

☐ because this figure better characterizes the invention.

☐ None of the figures.

#### NOTES TO FORM PCT/ISA/220 (continued)

The following examples illustrate the manner in which amendments must be explained in the accompanying letter:

1. [Where originally there were 48 claims and after amendment of some claims there are 51]:  
"Claims 1 to 29, 31, 32, 34, 35, 37 to 48 replaced by amended claims bearing the same numbers; claims 30, 33 and 36 unchanged; new claims 49 to 51 added."
2. [Where originally there were 15 claims and after amendment of all claims there are 11]:  
"Claims 1 to 15 replaced by amended claims 1 to 11."
3. [Where originally there were 14 claims and the amendments consist in cancelling some claims and in adding new claims]:  
"Claims 1 to 6 and 14 unchanged; claims 7 to 13 cancelled; new claims 15, 16 and 17 added." or  
"Claims 7 to 13 cancelled; new claims 15, 16 and 17 added; all other claims unchanged."
4. [Where various kinds of amendments are made]:  
"Claims 1-10 unchanged; claims 11 to 13, 18 and 19 cancelled; claims 14, 15 and 16 replaced by amended claim 14; claim 17 subdivided into amended claims 15, 16 and 17; new claims 20 and 21 added."

#### "Statement under Article 19(1)" (Rule 46.4)

The amendments may be accompanied by a statement explaining the amendments and indicating any impact that such amendments might have on the description and the drawings (which cannot be amended under Article 19(1)).

The statement will be published with the international application and the amended claims.

The statement should be brief, it should not exceed 500 words if in English or if translated into English:

It should not be confounded with and does not replace the letter indicating the differences between the claims as filed and as amended. It must be filed on a separate sheet and must be identified as such by a heading, preferably by using the words "Statement under Article 19(1)."

It should not contain any disparaging comments on the international search report or the relevance of citations contained in that report. Reference to citations, relevant to a given claim, contained in the international search report may be made only in connection with an amendment of that claim.

#### In what language ?

The amendments must be made in the language in which the international application is published. The letter and any statement accompanying the amendments must be in the same language as the international application if that language is English or French; otherwise, it must be in English or French, at the choice of the applicant.

#### Consequence if a demand for international preliminary examination has already been filed ?

If, at the time of filing any amendments under Article 19, a demand for international preliminary examination has already been submitted, the applicant must preferably, at the same time of filing the amendments with the International Bureau, also file a copy of such amendments with the International Preliminary Examining Authority (see Rule 62.2(a), first sentence).

#### Consequence with regard to translation of the international application for entry into the national phase ?

The applicant's attention is drawn to the fact that, where upon entry into the national phase, a translation of the claims as amended under Article 19 may have to be furnished to the designated/elected Offices, instead of, or in addition to, the translation of the claims as filed.

For further details on the requirements of each designated/elected Office, see Volume II of the PCT Applicant's Guide.

What Is Claimed Is:

1. A composition for lubricating metallic work pieces comprising:

(a) an oil having a viscosity of about 75 cSt to about 160 cSt at 25°C;

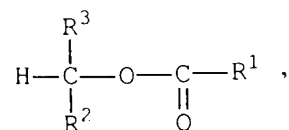
(b) free sulfur in an amount sufficient to provide lubrication, and

(c) a metal corrosion inhibitor to prevent corrosion of said work pieces;

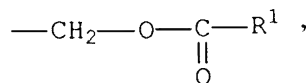
wherein said lubrication is demonstrated by a Falex reference load of greater than about 4,500 pounds force and by a Falex reference wear of less than ten teeth.

2. The composition of Claim 1, wherein said composition is a metalworking composition.

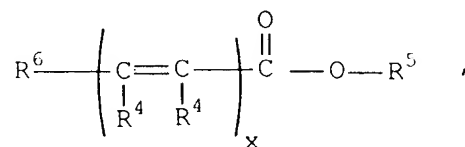
3. The composition of Claim 1, wherein said metal corrosion inhibitor is a fatty oil selected from the group consisting of a glyceride, an ester of a carboxylic acid, and combinations thereof, wherein said glyceride is represented by the formula of



wherein R<sup>1</sup> is a saturated or unsaturated C<sub>3</sub> to C<sub>24</sub> aliphatic hydrocarbon, and R<sup>2</sup> or R<sup>3</sup> is hydrogen or

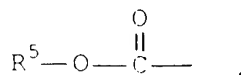


wherein R<sup>1</sup> is as defined above, and said ester is represented by the formula of



wherein R<sup>4</sup> is hydrogen or a saturated or unsaturated C<sub>3</sub> to C<sub>12</sub> aliphatic hydrocarbon, X is 1, 2 or 3, R<sup>5</sup> is a saturated or

unsaturated C<sub>3</sub> to C<sub>24</sub>, aliphatic hydrocarbon, and R<sup>6</sup> is represented by the formula of



wherein R<sup>5</sup> is as defined above.

4. The composition of Claim 3, wherein said fatty oil is about 5 to about 30 volume percent based on said composition.

5. The composition of Claim 1, wherein said sulfur is present in amounts of from about 0.4 to about 12 percent by weight of said composition.

6. The composition of Claim 1, wherein said composition when maintained at 100°C for 2 hours has a copper strip corrosion classification from about 1a to about 3b.

9. The composition of Claim 1, wherein said composition has a Four-Ball wear scar diameter of less than about 0.07 mm.

10. The composition of Claim 1, wherein the metallic work pieces are nonferrous metallic work pieces.

11. A composition for lubricating nonferrous metallic work pieces comprising:

(a) an oil having a viscosity suitable for heavy duty metalworking operations; and

(b) free sulfur being present in amounts of about 0.4 percent to about 12 percent by weight of said composition;

wherein said composition does not corrode said nonferrous work pieces.

12. The composition of Claim 11, wherein said sulfur is not chemically bound to molecules in said oil.

13. The composition of Claim 11, wherein said composition when maintained at 100°C for 2 hours has a copper strip corrosion classification of about 1a to about 2a.



18. The composition of Claim 11, wherein said composition has a viscosity of about 75 cSt to about 160 cSt at 25°C.

19. The composition of Claim 11, further comprising from about 0.0 to 4.0 weight percent chemically bound sulfur.

20. A method of making a composition which provides non-corrosive lubrication to nonferrous metalworking processes comprising:

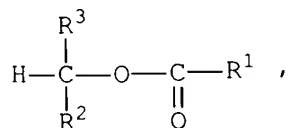
selecting a base oil having a viscosity of about 75 cSt to about 160 cSt at 25°C;

incorporating chemically unbound sulfur to said base oil to provide an extreme pressure lubricant, wherein the chemically unbound sulfur is incorporated in an amount from about 0.4 to about 12 weight percent of said composition; and

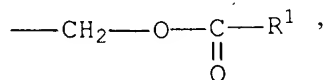
further incorporating a fatty oil to inhibit nonferrous metal corrosion.

21. The method of Claim 20, wherein said composition has a Falex reference wear of less than ten teeth.

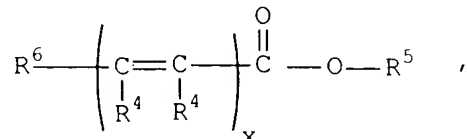
22. The method of Claim 20, wherein said fatty oil is selected from the group consisting of a glyceride, an ester of a carboxylic acid, and combinations thereof, wherein said glyceride is represented by the formula of



wherein R<sup>1</sup> is a saturated or unsaturated C<sub>3</sub> to C<sub>24</sub> aliphatic hydrocarbon and R<sup>2</sup> or R<sup>3</sup> is hydrogen or

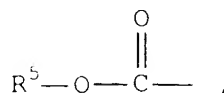


wherein R<sup>1</sup> is as defined above, and said ester is represented by the formula of



wherein R<sup>4</sup> is hydrogen or a saturated or unsaturated C<sub>3</sub> to C<sub>12</sub> aliphatic hydrocarbon, X is 1, 2 or 3, R<sup>5</sup> is a saturated or

unsaturated C<sub>3</sub> to C<sub>24</sub> aliphatic hydrocarbon, and R<sup>6</sup> is represented by the formula of



wherein R<sup>5</sup> is as defined above, and is combined into said composition in an amount from about 5 to about 30 volume percent based on the total composition and said fatty oil.

23. The method of Claim 20, further comprising incorporating from about 0.0 to 4.0 weight percent chemically bound sulfur.

24. A method of providing noncorrosive lubrication to the metalworking of a nonferrous metal part comprising:

providing a composition which includes a base oil having a viscosity of about 75 cSt to about 160 cSt at 25°C and free sulfur present in amounts sufficient to provide extreme pressure lubrication of a Falex reference load of greater than about 4,500 pounds force; and

applying said composition to the metal work part and/or a metal work tool during the metalworking process.

filed on  
5 March

What Is Claimed Is:

1. A composition for lubricating metallic work pieces comprising:

(a) an oil having a viscosity of about 75 cSt to about 160 cSt at 25°C;

(b) free sulfur in an amount sufficient to provide lubrication; and

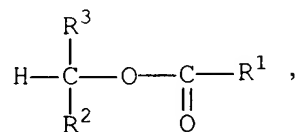
(c) a metal corrosion inhibitor to prevent corrosion of said work pieces,

wherein lubrication is demonstrated by measurements selected from the group consisting of Falex reference wear, Falex reference load, Four-Ball wear scar diameter, and combinations thereof.

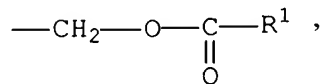
2. The composition of Claim 1, wherein said composition is a metalworking composition.

3. The composition of Claim 1, wherein said metal corrosion inhibitor is a fatty oil selected from the group consisting of a glyceride, an ester of a carboxylic acid, and combinations thereof,

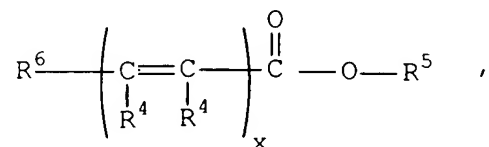
wherein said glyceride is represented by the formula of



wherein R<sup>1</sup> is a saturated or unsaturated C<sub>3</sub> to C<sub>24</sub> aliphatic hydrocarbon, and R<sup>2</sup> or R<sup>3</sup> is hydrogen or

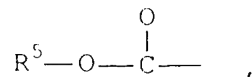


wherein R<sup>1</sup> is as defined above, and said ester is represented by the formula of



wherein R<sup>4</sup> is hydrogen or a saturated or unsaturated C<sub>3</sub> to C<sub>12</sub> aliphatic hydrocarbon, X is 1, 2 or 3, R<sup>5</sup> is a saturated or

unsaturated C<sub>3</sub> to C<sub>24</sub> aliphatic hydrocarbon, and R<sup>6</sup> is represented by the formula of



wherein R<sup>5</sup> is as defined above.

4. The composition of Claim 1, wherein said fatty oil is about 5 to about 30 volume percent based on said composition.

5. The composition of Claim 1, wherein said sulfur is present in amounts of from about 0.4 to about 12 percent by weight of said composition.

6. The composition of Claim 1, wherein said composition when maintained at 100°C for 2 hours has a copper strip corrosion classification from about 1a to about 3b.

7. The composition of Claim 1, wherein said composition has a Falex reference wear of less than ten teeth.

8. The composition of Claim 1, wherein said composition has a Falex reference load of greater than about 4,500 pounds force.

9. The composition of Claim 1, wherein said composition has a Four-Ball wear scar diameter of less than about 0.07 mm.

10. The composition of Claim 1, wherein the metallic work pieces are nonferrous metallic work pieces.

11. A composition for lubricating nonferrous metallic work pieces comprising:

(a) an oil having a viscosity suitable for heavy duty metalworking operations; and

(b) sulfur being present in amounts of about 0.4 percent to about 12 percent by weight of said composition; wherein said composition does not corrode said nonferrous work piece.

12. The composition of Claim 11, wherein said sulfur is not chemically bound to molecules in said oil.

13. The composition of Claim 11, wherein said composition when maintained at 100°C for 2 hours has a copper strip corrosion classification of about 1a to about 2a.

18. The composition of Claim 11, wherein said composition has a viscosity of about 75 cSt to about 160 cSt at 25°C.

19. The composition of Claim 11, further comprising from about 0.0 to 4.0 weight percent chemically bound sulfur.

20. A method of making a composition which provides non-corrosive lubrication to metalworking processes comprising:

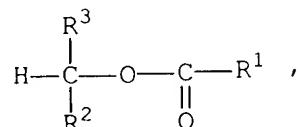
selecting a base oil having a viscosity of about 75 cSt to about 160 cSt at 25°C;

incorporating chemically unbound sulfur to said base oil to provide an extreme pressure lubricant; and

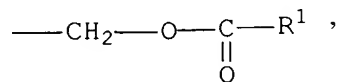
further incorporating a fatty oil to inhibit metal corrosion.

21. The method of Claim 20, wherein said composition has a Falex reference wear of less than ten teeth.

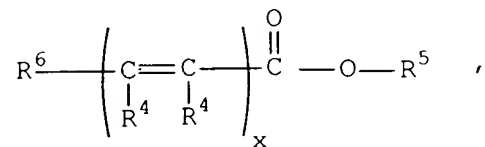
22. The method of Claim 20, wherein said fatty oil is selected from the group consisting of a glyceride, an ester of a carboxylic acid, and combinations thereof, wherein said glyceride is represented by the formula of



wherein R<sup>1</sup> is a saturated or unsaturated C<sub>3</sub> to C<sub>24</sub> aliphatic hydrocarbon and R<sup>2</sup> or R<sup>3</sup> is hydrogen or

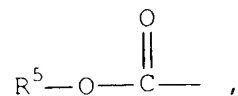


wherein R<sup>1</sup> is as defined above, and said ester is represented by the formula of



wherein R<sup>4</sup> is hydrogen or a saturated or unsaturated C<sub>3</sub> to C<sub>12</sub> aliphatic hydrocarbon, X is 1, 2 or 3, R<sup>5</sup> is a saturated or

unsaturated C<sub>3</sub> to C<sub>24</sub> aliphatic hydrocarbon, and R<sup>6</sup> is represented by the formula of



wherein R<sup>5</sup> is as defined above, and is combined into said composition in an amount from about 5 to about 30 volume percent based on the total composition and said fatty oil.

23. The method of Claim 20, further comprising incorporating from about 0.0 to 4.0 weight percent chemically bound sulfur.

24. A method of providing noncorrosive lubrication to the metalworking of nonferrous metal parts comprising:

providing a composition which includes a base oil having a viscosity of about 75 cSt to about 160 cSt at 25°C and free sulfur present in amounts sufficient to provide extreme pressure lubrication; and

applying said composition to the metal work piece and/or metal work tool during the metalworking process.

ABSTRACT OF THE DISCLOSURE

Non-staining, active metal-working compositions are disclosed. The compositions contain active sulfur to provide extreme pressure properties for metal-working fluids. A metal  
5 corrosion inhibitor is disclosed that reduces the corrosivity of free sulfur on non-ferrous metallic objects.